
**Rubber, vulcanized — Determination of
temperature rise and resistance to fatigue
in flexometer testing —**

**Part 4:
Constant-stress flexometer**

*Caoutchouc vulcanisé — Détermination de l'élévation de température
et de la résistance à la fatigue dans les essais aux flexomètres —*

Partie 4: Flexomètre à contrainte constante



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

ISO 4666-4 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

ISO 4666 consists of the following parts, under the general title *Rubber, vulcanized — Determination of temperature rise and resistance to fatigue in flexometer testing*:

- *Part 1: Basic principles*
- *Part 2: Rotary flexometer*
- *Part 3: Compression flexometer*
- *Part 4: Constant-stress flexometer*

Introduction

This part of ISO 4666 describes a method of compression flexometer testing with constant-stress dynamic loading. The features and usefulness of constant-stress flexometer testing are as follows:

- a) In order to exactly simulate the behaviour of a rubber product in use, an important consideration is where the temperature is measured. The constant-stress flexometer measures the temperature directly at the centre of the inside of the test piece (the source of heat generation), using a device as shown in Figure 4 of this part of ISO 4666, while in Part 3 of this International Standard the temperature is measured on the surface of the test piece.
- b) A servo control system based on real-time feedback of the strain or stress is used to enable the measurement of dynamic properties (viscoelastic parameters) of the rubber as a function of time during the test run.
- c) The accumulation of feedback information allows the detection of an initial stage, or the first signs of breakdown due to heat generation, which was once thought to be very difficult.

It has been reported^[1] how well the rise in tyre temperature correlates with the temperature rise in the constant-stress flexometer test in comparison with the result from the method in Part 3 of this International Standard.

The International Organization for Standardization (ISO) draws attention to the fact that it is claimed that compliance with this document may involve the use of a patent concerning the flexometer specified in Clause 5.

ISO takes no position concerning the evidence, validity and scope of this patent right.

The holder of this patent right has assured ISO that he is willing to negotiate licences under reasonable and non-discriminatory terms and conditions with applicants throughout the world. In this respect, the statement of the holder of this patent right is registered with ISO. Information may be obtained from:

Bridgestone Corporation, 3-1-1 Ogawahigashi-Cho, Kodaira-Shi, Tokyo 187-8531, Japan.

Attention is drawn to the possibility that some elements of this document may be the subject of patent rights other than those identified above. ISO shall not be held responsible for identifying any or all such patent rights.

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Rubber, vulcanized — Determination of temperature rise and resistance to fatigue in flexometer testing —

Part 4: Constant-stress flexometer

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This part of ISO 4666 specifies a constant-stress flexometer test for the determination of the temperature rise and resistance to fatigue of vulcanized rubbers.

Many rubber products, such as tyres and belts, are tested by subjecting them to an oscillating load with a constant peak stress amplitude. In order to obtain good correlation between accelerated tests and in-service exposure of these products, this part of ISO 4666 gives instructions for carrying out measurements under such conditions.

This method is not recommended for rubber having a hardness greater than 85 IRHD.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 48, *Rubber, vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD)*

ISO 4664-1, *Rubber, vulcanized or thermoplastic — Determination of dynamic properties — Part 1: General guidance*

ISO 4666-1, *Rubber, vulcanized — Determination of temperature rise and resistance to fatigue in flexometer testing — Part 1: Basic principles*

ISO 4666-3, *Rubber, vulcanized — Determination of temperature rise and resistance to fatigue in flexometer testing — Part 3: Compression flexometer*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4664-1 and ISO 4666-1 apply.

4 Principle

A cylindrical test piece is subjected to dynamic loading with constant peak stress cycles in compression superimposed on a static prestress.

The temperature rise of the test piece is measured, and the fatigue life of the test piece is given by the number of cycles, or the test time, until breakdown occurs. The change in height (creep) and dynamic properties are also measured as a function of time, and the compression set is measured at the end of the test.

5 Apparatus

The apparatus is shown schematically in Figure 1, and an example is shown in Figure 2.

5.1 Anvils

A pair of anvils (upper and lower) support the test piece. The lower anvil is connected to an oscillator to apply static and dynamic compression deformation to the test piece, and the upper anvil transmits the static and dynamic compression loads, via a shaft, to a load detector. The parts of the upper and lower anvils which come in contact with the test piece shall be made of a heat-insulating material of thermal conductivity 0,28 W/(m·K) maximum. A hole shall be provided in the centre of the upper anvil for insertion of a needle-type thermometer for measuring the temperature inside the test piece. An example of upper and lower anvil construction is shown in Figure 3.

5.2 Oscillator

The oscillator used to apply static and dynamic compression loads to the test piece shall have a capacity of at least 2 kN and be capable of applying an oscillating force of 0,75 kN peak amplitude at 50 Hz.

A hydraulic servo-control system is preferably used to control the oscillator.

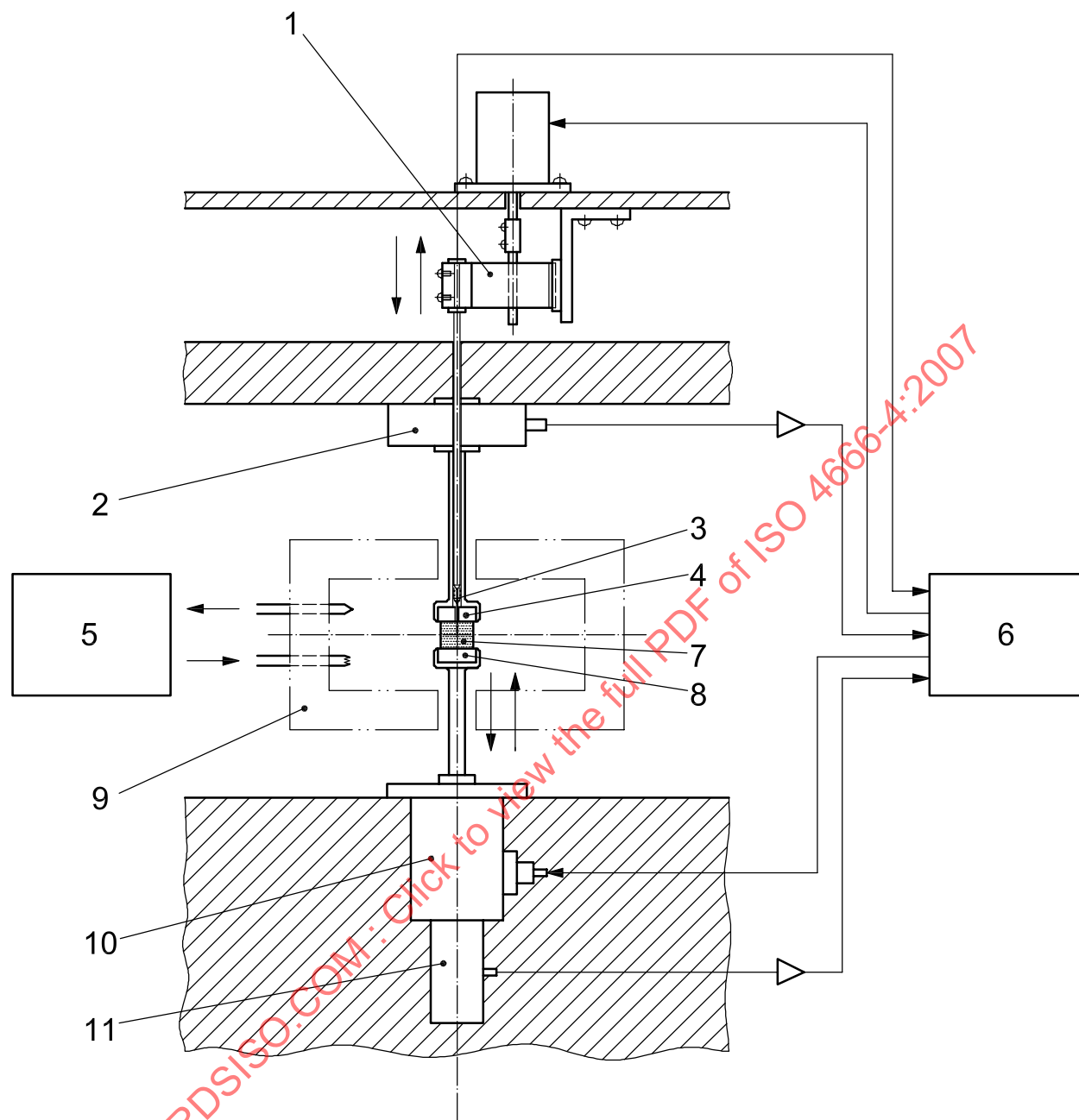
The maximum stroke is preferably 20 mm to 25 mm.

5.3 Displacement detector

The displacement detector shall be capable of measuring the motion of the lower anvil (the deformation of the test piece in compression) to within 0,01 mm, and shall have a response time suitable for the maximum frequency used.

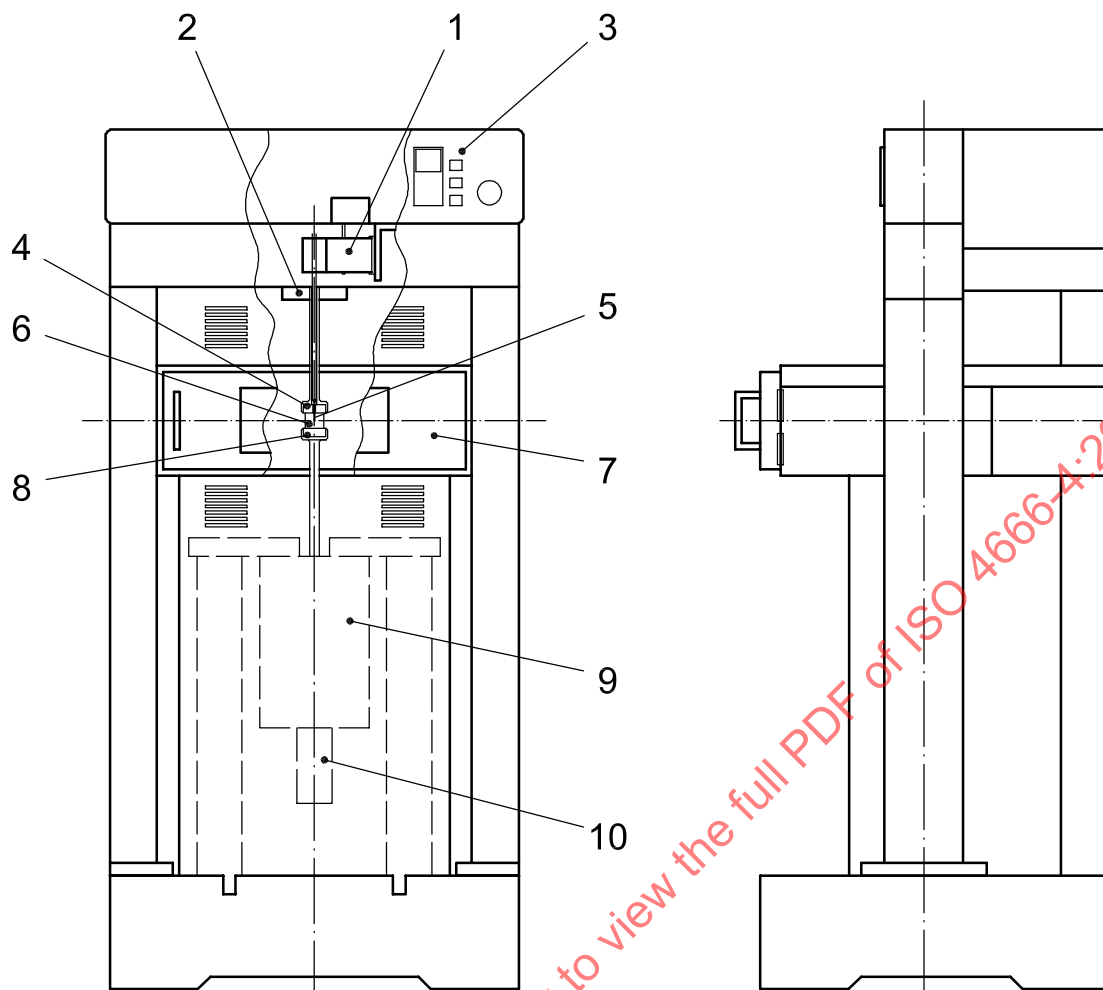
5.4 Load detector

The load detector shall be capable of measuring the compression load up to a maximum of 2,0 kN in 5 N increments, shall have a response time suitable for the maximum frequency used, and shall have a high natural frequency.


Key

- 1 position controller
- 2 load detector
- 3 needle-type temperature detector
- 4 upper anvil
- 5 temperature controller
- 6 computer control unit
- 7 test piece
- 8 lower anvil
- 9 heating chamber
- 10 oscillator
- 11 displacement detector

Figure 1 — Principle and fundamental structure of a constant-stress flexometer

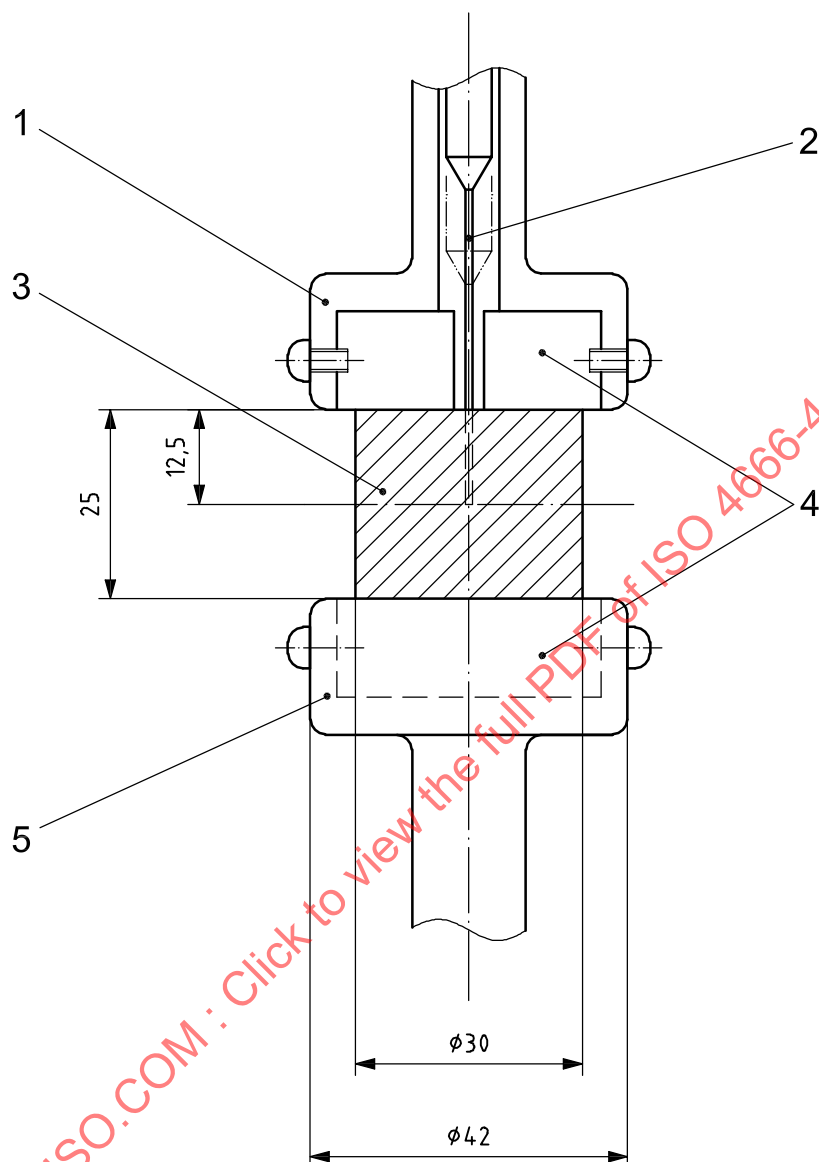


Key

- 1 position controller
- 2 load detector
- 3 temperature controller
- 4 upper anvil
- 5 needle-type temperature detector
- 6 test piece
- 7 heating chamber
- 8 lower anvil
- 9 oscillator
- 10 displacement detector

Figure 2 — An example of a constant-stress flexometer

Dimensions in millimetres

**Key**

- 1 upper anvil
- 2 needle-type temperature detector
- 3 test piece
- 4 thermal insulator
- 5 lower anvil

Figure 3 — An example of upper and lower anvils for a constant-stress flexometer

5.5 Heating chamber and temperature controller

The temperature of the heating chamber shall be set at a temperature within the range 40 °C to 100 °C as specified in ISO 23529, and be controlled to within ± 1 °C. The temperature in the chamber shall be measured at positions 6 mm to 9 mm away from the end of each anvil and also midway between the upper and lower anvils. A temperature sensor wire at least 100 mm in length shall be inserted into the chamber.

A grid shelf on which to condition test pieces should preferably be installed in the chamber at a similar height to that of the lower anvil, although conditioning of test pieces may also be carried out in another heating chamber.

5.6 Needle-type temperature detector

A needle-type temperature detector with a diameter at the tip of 1,0 mm and resolution of $\pm 0,5$ °C shall be used.

An example of a needle-type temperature detector is shown in Figure 4.

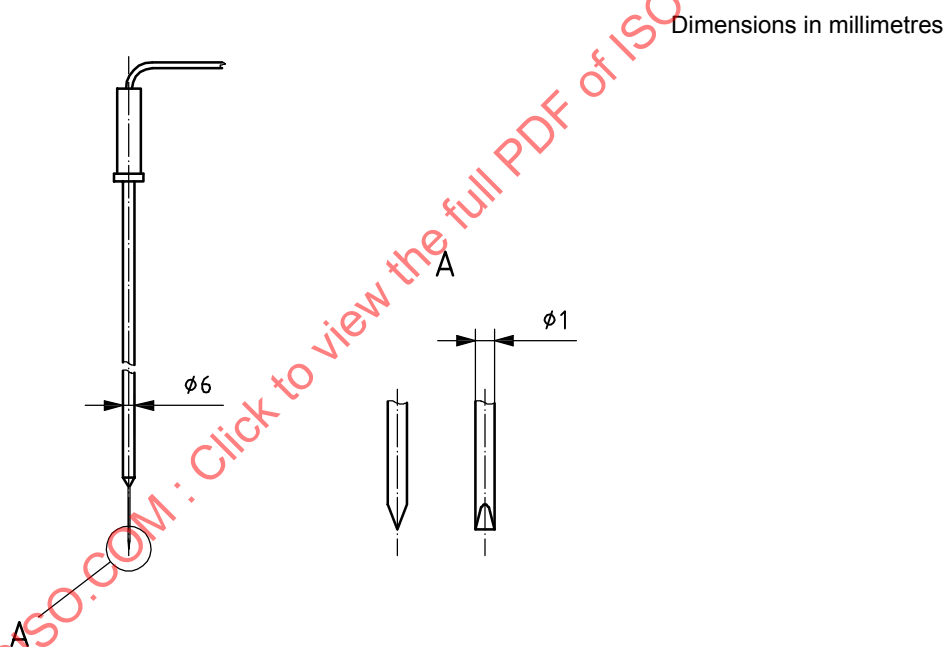


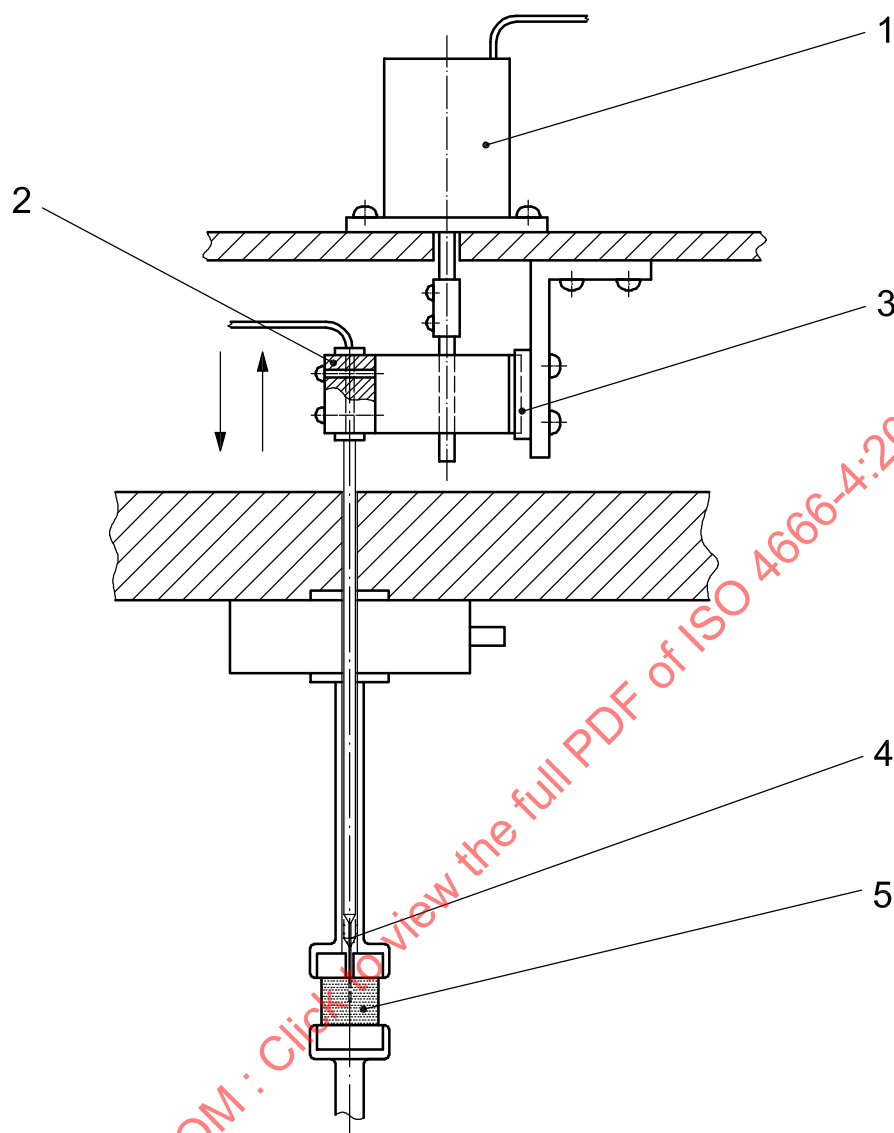
Figure 4 — Example of a needle-type temperature detector

5.7 Temperature-detector position controller

The position controller shall be capable of adjusting the position of the needle-type temperature detector using the feedback data on the test piece height sent from the displacement detector through the computer control unit during the test in real time.

NOTE The height of a test piece refers to the average value of the maximum height and the minimum height in one cycle of a compression-oscillating test piece. In general, this value decreases gradually during the test due to creep of the test piece.

An example of a temperature-detector position controller is shown in Figure 5.

**Key**

- 1 stepping motor
- 2 clamp
- 3 guide
- 4 needle-type temperature detector
- 5 test piece

Figure 5 — Example of a temperature-detector position controller

5.8 Computer control unit

The computer control unit shall be capable of the following:

- a) controlling the action of the oscillator so that the static compression stress applied to the test piece always coincides with the value specified in the test conditions;
- b) controlling the action of the oscillator so that the amplitude of the dynamic stress applied to the test piece always coincides with the value specified in the test conditions (constant-stress control);
- c) recording and displaying the temperature at the centre of the test piece detected by the needle-type temperature detector;
- d) calculating, recording and displaying the creep of the test piece from the values measured by the displacement detector;
- e) (when determining the fatigue life from dynamic properties) calculating, recording and displaying the dynamic properties of the normal storage modulus E' , normal loss modulus E'' and tangent of the loss angle ($\tan \delta$) from the measured parameters (see 8.3.4) fed back from the sensors in real time, these values being preferably calculated at 1 s intervals;
- f) ending the test at the time specified in the test conditions or at the time when the recorded values reach specified limits.

5.9 Measuring gauge

The gauge for measuring the height and diameter of test pieces shall conform to the requirements of ISO 23529. A dial gauge having a circular foot probe of diameter 10 mm and exerting a pressure of 22 kPa $\pm$ 5 kPa is suitable.

6 Test piece

The test piece, prepared from vulcanized rubber, shall be cylindrical in shape, having a diameter of 30,00 mm $\pm$ 0,30 mm and a height of 25,00 mm $\pm$ 0,25 mm.

The standard method of preparing the test piece shall be direct moulding of the cylinder. It is suggested, for purposes of uniformity and closer tolerances in the moulded test piece, that the dimensions of the mould be specified and shrinkage compensated for therein.

NOTE A plate cavity of diameter 30,40 mm $\pm$ 0,05 mm and depth 25,40 mm $\pm$ 0,05 mm, having overflow cavities at both top and bottom when assembled with two end plates, represents one such type of mould.

7 Test conditions

The conditions specified in Table 1 or Table 2 are normally used in tests with the constant-stress flexometer.

The dynamic-load amplitude shall be less than the static load.

Table 1 — Test conditions for measurement of temperature rise

Conditions	Nominal value	Range
Chamber temperature	Standard laboratory temperature [(40 ± 1) °C or (100 ± 1) °C]	—
Static load	600 N	250 N to 900 N
Dynamic-load amplitude	400 N	200 N to 700 N
Frequency	10 N	5 Hz to 30 Hz

Table 2 — Test conditions for detection of fatigue breakdown

Conditions	Nominal value	Range
Chamber temperature	(40 ± 1) °C or (100 ± 1) °C	—
Static load	680 N	510 N to 950 N
Dynamic-load amplitude	600 N	500 N to 750 N
Frequency	30 Hz	20 Hz to 50 Hz

The normal test duration is 25 min for the measurement of temperature rise. However, if required, a longer test duration may be selected.

For the detection of fatigue breakdown, the test duration shall be the time until breakdown begins inside the test piece. If fatigue breakdown is not induced after 25 min, the test shall be repeated under more severe conditions. If breakdown occurs too quickly, the test shall be repeated under less severe conditions.

NOTE The method and conditions for detecting fatigue breakdown vary according to the type of product and the purpose of the test. Therefore, they cannot generally be specified. A general procedure for detecting fatigue breakdown automatically is given in 8.2.

8 Procedure

8.1 General test procedure

The test shall be carried out as follows:

- Measure the height of the test piece.
- To set up the test piece in its correct position, operate the oscillator in a manual mode, then move the lower anvil to the lowest position, place the test piece at the centre of the lower anvil, and move the lower anvil upwards until the upper surface of the test piece comes in contact, or almost in contact, with the upper anvil. At this time, do not apply a load of more than 5 N to the test piece and do not allow the clearance between the upper anvil and the upper surface of the test piece to be more than 0,5 mm. When the test is carried out at elevated temperature, first place the test piece on the grid shelf in the heating chamber and condition for at least 30 min.
- Using the position controller, insert the needle-type temperature detector at the centre of the upper surface of the test piece to a depth of 12,5 mm. Further, set the position controller such that the needle-type temperature detector is automatically controlled to remain at a depth of half of the average height of the test piece while the test piece height is decreasing due to creep.
- Move the lower anvil by operating the oscillator and compress the test piece until the specified static load is applied to the test piece. At this time, the position controller starts to move the needle-type temperature detector to adjust the depth to half of the reduced test piece height.

- e) 5 s to 10 s after the application of the static load to the test piece, when the temperature indicated by the needle-type temperature detector has stabilized, switch the oscillator operation to the automatic control mode to operate the lower anvil so that a dynamic load with amplitude and frequency set previously is applied to the test piece. Take this time as the start time of the test.
- f) During the test, maintain the conditions so that the average values of the static load and the amplitude of the dynamic load applied to the test piece coincide with the specified static load and the dynamic-load amplitude, respectively.
- g) Record and display the temperature and the creep of the test piece in the computer control unit.
- h) For determination of the fatigue life from dynamic properties, the cyclic load and displacement at the specified time interval are fed from the force and displacement sensors to the computer control unit which also calculates and displays the dynamic properties of normal storage modulus E' , normal loss modulus E'' and tangent of the loss angle $\tan \delta$. The specified time interval is preferably 1 s.

8.2 Determination of fatigue life

8.2.1 Practical method

To determine the fatigue life, continue the test until breakdown occurs. The fatigue life is expressed as the number of cycles N , or the time, to breakdown or failure of the test piece. Breakdown can be recognized by a significant change in the dynamic properties observed on the real-time display system, or by an irregularity in the temperature curve (a sudden temperature rise), or by a significant increase in creep. After ending the test, cut the test piece horizontally at mid-height and visually confirm and report the degree of damage (fine bubbles at the centre of the test piece, cracks or deterioration of rubber quality).

8.2.2 Automatic method

8.2.2.1 General

Since breakdown starting at the centre of the inside of the test piece is difficult to see, an indirect detection method is often more convenient. In a constant stress test, automatic detection of the beginning of breakdown can be achieved by considering the changes in the parameters monitored during the test.

8.2.2.2 Parameter for detecting breakdown

As the following parameters are continuously measured during the test in real time, breakdown can be recognized by following the variation in any of them:

- the temperature of the inside of the test piece θ ;
- the creep F ;
- the normal storage modulus E' ;
- the normal loss modulus E'' ;
- the tangent of the loss angle $\tan \delta$.

In the case of the temperature, creep and normal storage modulus of the test piece, fatigue breakdown has generally already developed considerably at the point in time when a noticeable change is shown, but these parameters are nevertheless applicable for detection of complete breakdown. Changes in normal loss modulus or tangent of the loss angle are well suited to detecting the initial stage of breakdown.

8.2.2.3 Determination of the criteria for breakdown

When detecting fatigue breakdown automatically, criteria for judging the condition of the test piece as observed after dissection are necessary, for example the number of bubbles appearing on the section and their size (diameter). The degree of breakdown can be divided into several levels. The criteria for the beginning of breakdown shall be determined in advance.

Since the criteria for breakdown are determined by the kind of product and the purpose of the test, they cannot be specified universally.

8.2.2.4 Determination of the point at which breakdown can be assumed to have occurred

If it is required for automatic detection of breakdown, any one of the parameters in 8.2.2.2 (or a combination of two or more parameters) may be used and, when the amount of change in the value, the rate of change with time or another form of change (hereafter referred to as the change in the parameter) reaches a specified level, the breakdown is considered to be detected, and the test is stopped. The following procedure can be used in order to determine experimentally the conditions for such detection of breakdown.

- a) Prepare samples of several materials (preferably at least five) with as wide a range of fatigue lives as possible, and prepare at least six test pieces from each sample. In the case where breakdown is to be detected by change in the normal loss modulus and in the tangent of the loss angle, samples with different dynamic characteristics are preferable.
- b) The static load and the dynamic-load amplitude equal to, or similar to, the service conditions in which the product will be used shall be selected. For an accelerated test, the values of test temperature and frequency shall be higher than those in the service conditions.
- c) Carry out a test with the test time being long enough to definitely cause breakdown. During this test, estimate the time to initiation of breakdown from the change in a continuously measured parameter. Repeat the test, but with a test time equal to the time to initiation of breakdown determined in the first test.
- d) After the second test, cut the test piece and compare the degree of damage with a previously determined level.
- e) Repeat the test, selecting a longer test time if the degree of damage at the level of breakdown detected automatically was insufficient, and by selecting a shorter time if excessive breakdown occurred.
- f) When the desired level of breakdown to be detected is obtained by repeating steps c) to e), record quantitatively details of the change of the measured parameter together with the test time.
- g) Repeat steps c) to f) for all the prepared samples.
- h) Obtain an experimental formula by analysing the results obtained and determine the condition for automatic detection of breakdown.

A new test piece is used each time for c) to f).

If breakdown is not generated even though the test is carried out for 25 min in step c), or if breakdown is generated too early, repeat the test with modified test conditions (see Clause 7).

In step f), in order to confirm the result, the test is preferably carried out under the same conditions using at least two test pieces.

8.3 Determination of changes in specific parameters

8.3.1 Temperature rise

To determine the temperature rise, measure the internal temperature of the test piece over the test duration.

The temperature rise is given by the equation

$$\Delta\theta = \theta_{25} - \theta_0 \quad (1)$$

where

$\Delta\theta$ is the temperature rise, in °C, of the test piece;

θ_{25} is the temperature, in °C, of the test piece after 25 min of testing;

θ_0 is the temperature, in °C, of the test piece at the beginning of the test.

NOTE The temperatures θ_0 and θ_{25} are measured at the centre of the inside of the test piece.

8.3.2 Creep

To determine the creep, measure the test piece height 6 s after the start of cyclic loading and then after a specified test duration.

The creep is defined by the equation

$$F_t = \frac{h_6 - h_t}{h_0} \times 100 \quad (2)$$

where

F_t is the creep, in %, after the test duration t ;

h_6 is the test piece height, in mm, determined 6 s after the start of cyclic loading;

h_t is the test piece height, in mm, determined after the test duration t ;

h_0 is the original height of the test piece, in mm, in the unloaded condition.

Equation (2) can also be expressed in the form given in Equation (3):

$$F_t = \frac{h_0 - h_t}{h_0} \times 100 - \frac{h_0 - h_6}{h_0} \times 100 = F_{(t)} - F_{(6)} \quad (3)$$

In Equation (3), $F_{(6)}$ indicates the initial creep. As F_t alone does not include any information on the initial creep, the pair F_t and $F_{(6)}$ or the pair $F_{(t)}$ and $F_{(6)}$ shall be used to express the creep characteristics.

The test piece height shall be measured as previously described. For the original height h_0 , the nominal value $h_0 = 25$ mm may be used, provided it has been confirmed that any difference is within a tolerance range of $\pm 0,2$ mm.

Append the test duration, in minutes, to the result in parentheses.

8.3.3 Compression set

At the end of the test, remove the test piece from the apparatus and, after conditioning it at standard laboratory temperature for 1 h, measure the height h_e .

The compression set is given by the equation

$$S = \frac{h_0 - h_e}{h_0} \times 100 \quad (4)$$

where

S is the compression set, in %;

h_0 is the original height of the test piece, in mm, in the unloaded condition;

h_e is the final height of the test piece, in mm, after conditioning for 1 h in the unloaded condition.

8.3.4 Dynamic properties

The dynamic properties of normal storage modulus E' , normal loss modulus E'' and tangent of the loss angle $\tan\delta$ are calculated by the computer control unit using equations specified in ISO 4664-1 from the measured compression load and the deformation of the test piece as follows:

$$|E^*| = \frac{F_0/A}{x_0/h_0} \quad (5)$$

$$E' = |E^*| \cos\delta \quad (6)$$

$$E'' = |E^*| \sin\delta \quad (7)$$

$$\tan\delta = \frac{E''}{E'} \quad (8)$$

where

$|E^*|$ is the absolute value of the normal modulus, in MPa;

F_0 is the load amplitude, in N, from a force sensor;

A is the cross-sectional area of the test piece, in mm²;

x_0 is the maximum deflection amplitude, in mm, from a displacement sensor;

h_0 is the original height of the test piece in the unloaded condition, in mm;

E' is the normal storage modulus, in MPa;

E'' is the normal loss modulus, in MPa;

$\tan\delta$ is the tangent of the loss angle;

δ is the loss angle, in rad, measured as the difference in phase angle between load and displacement.

9 Precision

See Annex A.

10 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- b) sample details:
 - 1) a full description of the sample and its origin,
 - 2) details of the compound and of the vulcanizing conditions, if known;
- c) test pieces:
 - 1) the method of preparation, for example whether moulded or cut or taken from finished products,
 - 2) the original height and, in the case of deviations from the standard dimensions, the diameter,
 - 3) the hardness;
- d) test details:
 - 1) the static load (or stress), in N (or MPa),
 - 2) the dynamic-load (or stress) amplitude, in N (or MPa),
 - 3) the frequency, in Hz,
 - 4) the heating-chamber temperature, in °C,
 - 5) any deviations from the procedures specified in this International Standard;
- e) test results:
 - 1) for the measurement of temperature rise: the test duration, the individual values and the mean value,
 - 2) for the measurement of creep: the test duration, the individual values and the mean value,
 - 3) for the measurement of compression set: the test duration, the individual values and the mean value,
 - 4) for the measurement of fatigue resistance: the number of test pieces used, the criterion for fatigue breakdown, and the time or the number of cycles to this selected breakdown point, expressed as individual values and the mean value,
 - 5) the dynamic properties, if desired, expressed as individual values and the mean value:
 - normal storage modulus E' ,
 - normal loss modulus E'' ,
 - tangent of the loss angle $\tan \delta$,
 - 6) details of the visual observations made on the cut test pieces after testing;
- f) the date of the test.